An Alternative Technique for Determining Gravimetric Particle Mass Deposition on Filter Substrate: The Particle Extraction Method

~~CHARITY GARLAND~~

MADELEINE ROSSANESE

BERKELEY AIR MONITORING GROUP

ETHOS 2019

Objectives

- Understand standard gravimetric particulate matter (PM) measurements
- Identify the limitations with standard gravimetric PM measurements
- Understand extraction method particulate matter measurements
- Identify the limitations of the extraction method for PM measurements

Background

- Gravimetric filter analysis, employing a pump and filter system, is the gold standard method for measuring the mass concentration of particulate matter (PM) in air
- Uses high precision (0.1 - 1 µg resolution) mass measurements of filters before and after sample collection.
- Scales must be operated in a highly controlled environment that is clean, demonstrates low static and vibration, and is temperature and humidity stable
- Field blanks are typically used to account for changes in mass unrelated to sampling (e.g. contamination/mass loss from handling, transport, etc.)

(Balakrishnan et al. , 2015; Health and Safety Executive (HSE), 1997; Rosa et al. , 2014)

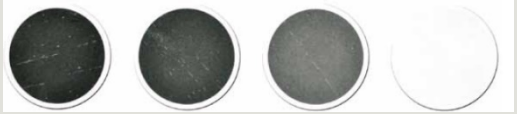
Standard Gravimetric Method

https://www.mt.com

Pre-weigh

Transport to the field site

Sampling



Transport to the lab

https://www.mt.com

Post-weigh

Post-Weight – Pre-Weight = Mass Deposited

Mass Deposited / Volume of Air = Average PM concentration ($\mu g/m^3$)

Standard Gravimetric Limitations

- Conditions must be well-matched between pre- and post-weights, which may be separated by several months and up to years
- Filter data may be lost or compromised by incorrect mass measurements resulting from unstable scales, improper scale operation or calibration, or transcription errors
- Sample sets can be comprised of thousands of filters - some erroneous mass measurements are likely to occur.
- Post-weight measurements can be repeated if filters are stored properly, there is currently no method for recovering lost or compromised pre-weights

Extraction Method

Sampling

Transport to lab

Post-weight (x2)

https://www.mt.com

Extraction

Post-extraction Weight (pre-weight proxy)

https://www.mt.com

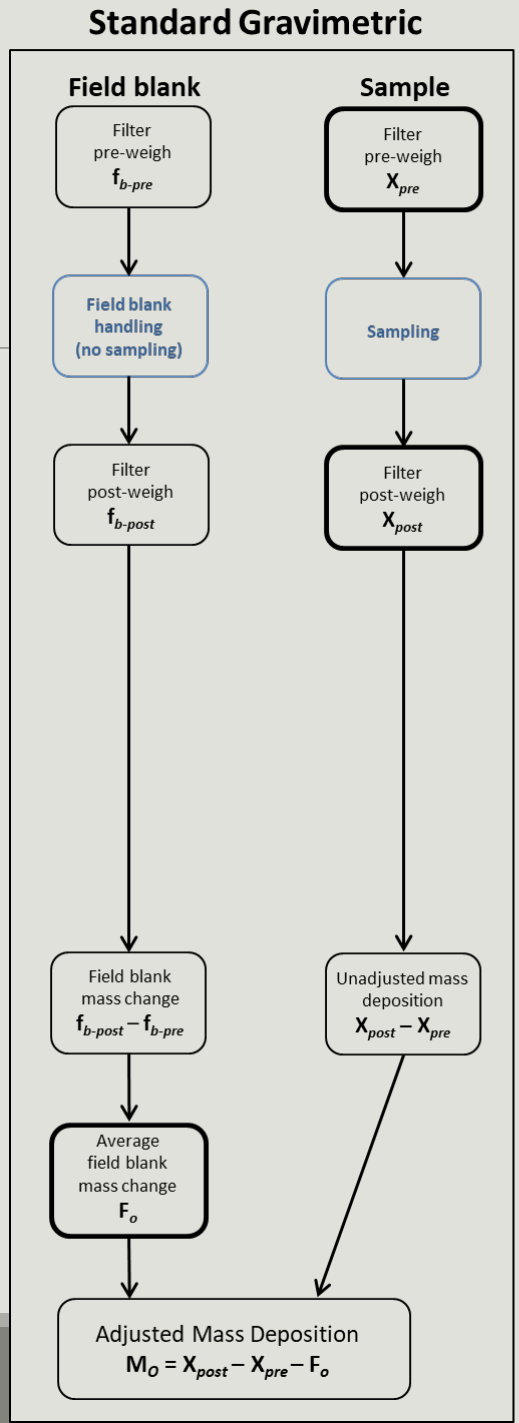
Post-Weight – Post-extraction = Mass Deposited

Mass Deposited / Volume of Air = Average PM concentration ($\mu g/m^3$)

Method Comparison

Sample Filters: Used to collect PM sample for concentration calculation

Field Blanks: Treated the same way as the sample filters, apart from sampling air. In the extraction method, field blanks also undergo the extraction process. Used to correct for unexpected changes in mass related to transport, handling, storage, and the extraction process.



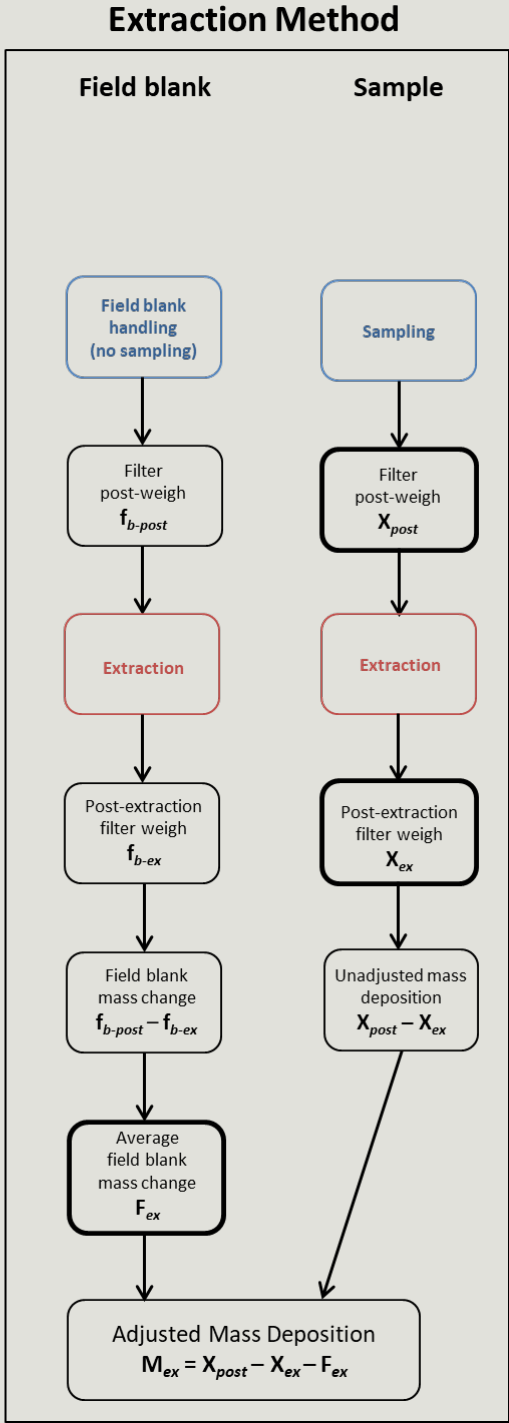


Image by Madeleine Rossanese

Study

Samples:

1. 265 sample PTFE filters, primarily wood smoke PM2.5 deposition. Six samples were excluded due to incorrect pre-sampling weights, evidenced by negative mass depositions. 28 filters were excluded that were below the extraction method's limit of detection of 60 µg
2. 21 field blanks (handled similarly to the sample filters but not used for sampling), including undergoing the extraction process,
3. Eight extraction blanks (filters taken directly from the package and not otherwise handled or sampled), and
4. Four lab-simulated filter samples

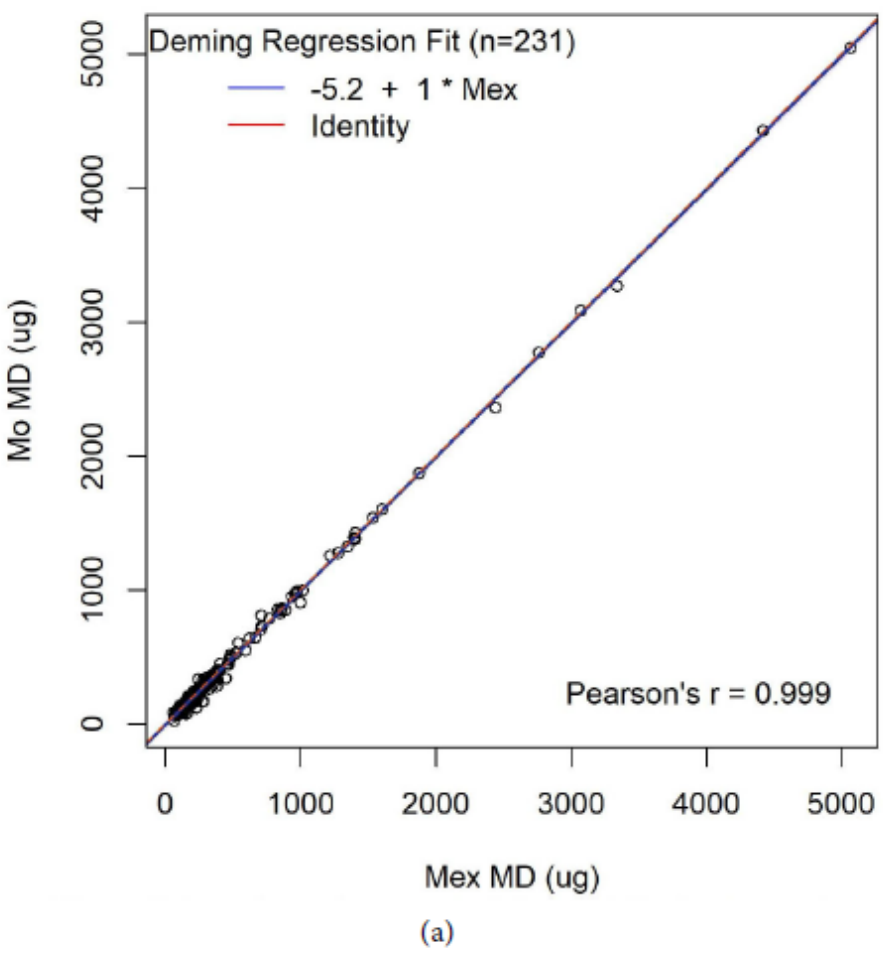
Limit of Detection

Extraction blanks were analyzed to determine the limit of detection (LoD) for this method using the commonly accepted calculation for LOD shown in the equation below (y_{LOD}).

$$Y_{LOD} = B_{ex} + 3 * \sigma_{Bex}$$

- Average mass change (Bex) = 21 µg for all extraction blanks (N = 8)
- Standard deviation of the extraction blank mass change (σBex) = 13 µg
- Y_{LoD} = 60 µg.

All samples with Mex below 60 µg were excluded from the following analysis (N = 28) for a total sample size of 231. Of these 28 filters, 11 were sample filters (4%) and 17 were blanks.



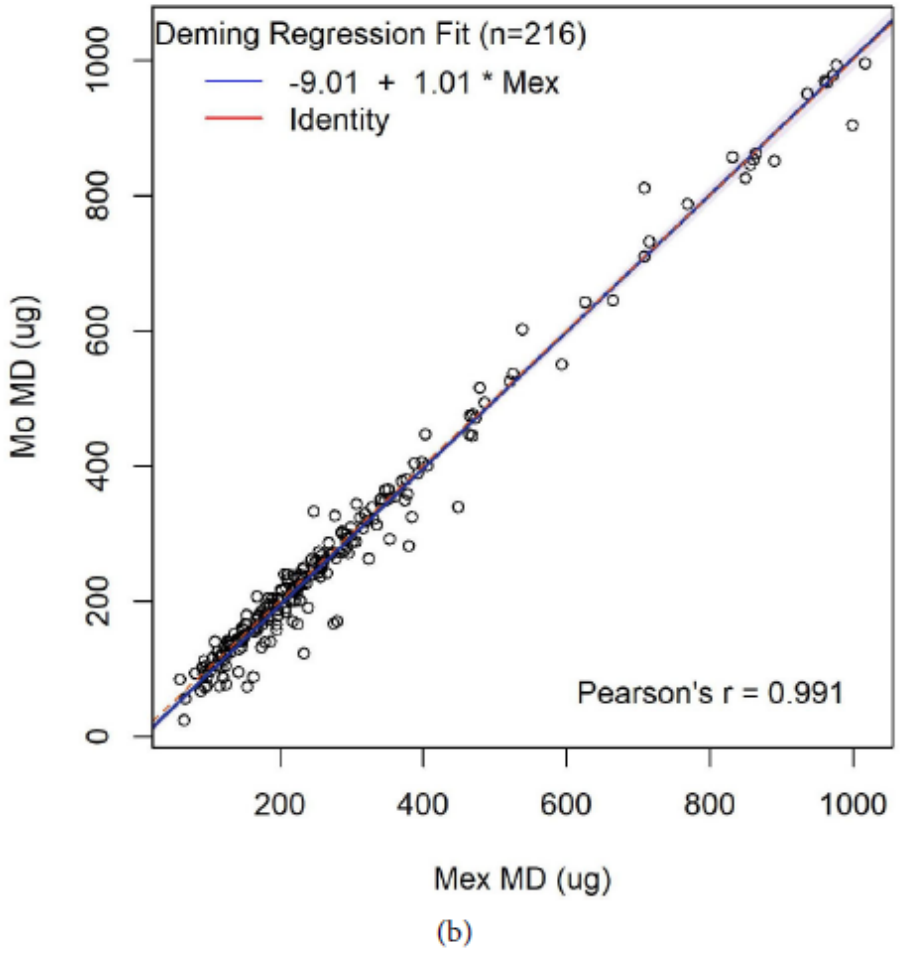


Figure 2. A Deming regression model relating original mass deposition from the standard gravimetric method (M_o) versus mass deposition determined from the extraction method (M_{ex}) for all filters (a) and for filters with an M_o of 1000 µg or less (b). The 95% confidence bounds are calculated with the bootstrap (quantile) method. The "Identity" line indicates equivalent methods.

(Deming, W.E., 1943)

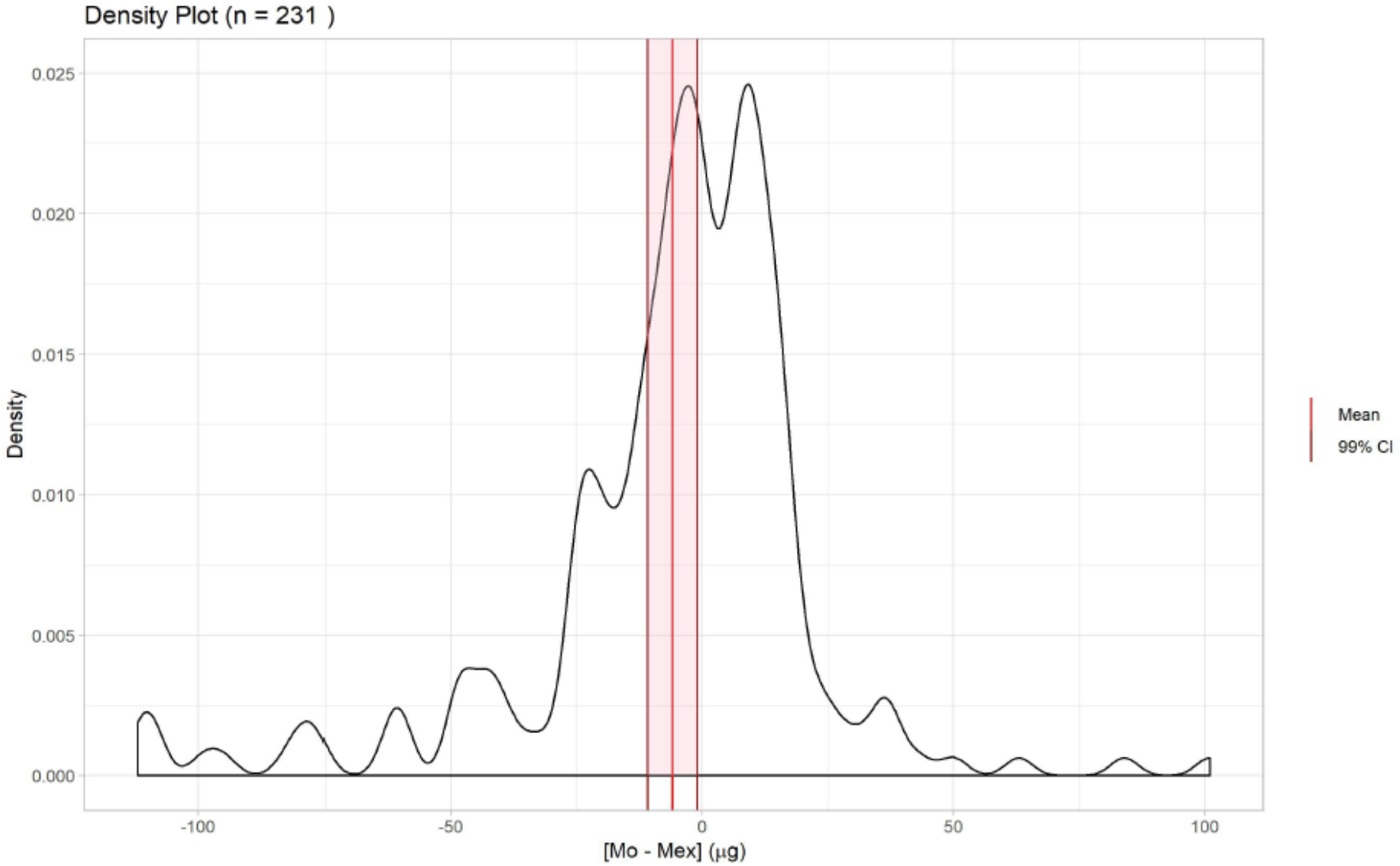


Figure 3. The mean mass deposition difference determined from field-blank adjusted original pre-sampling weight [M_o] and field-blank adjusted post-extraction weights [M_{ex}] is −5.8 ± 4.5. The distribution of the differences in mass deposition using the two methods is shown with the 99% confidence interval and mean indicated by the vertical lines.

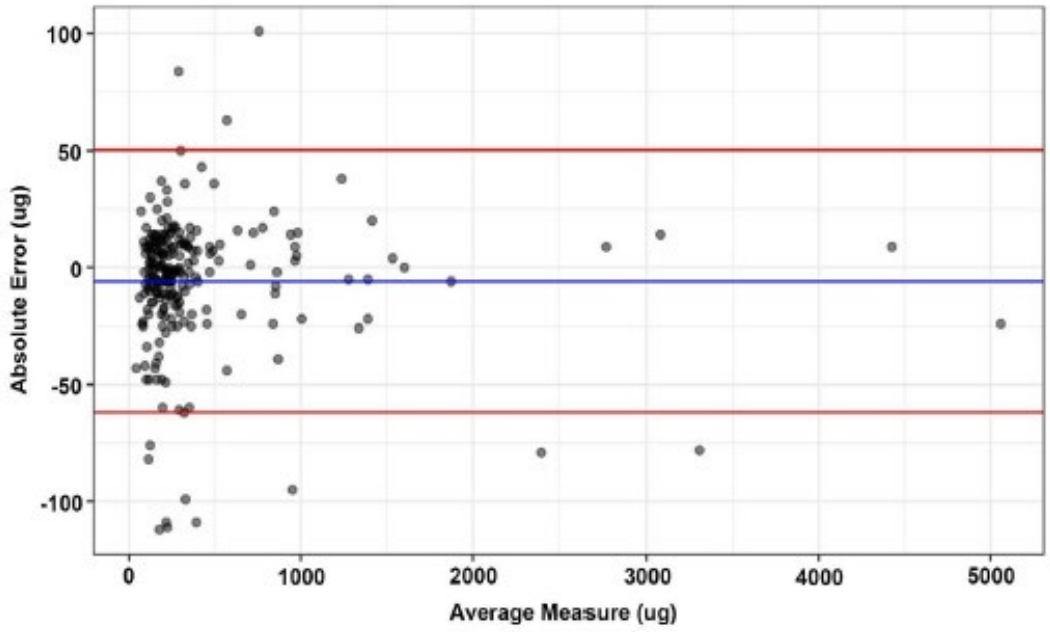
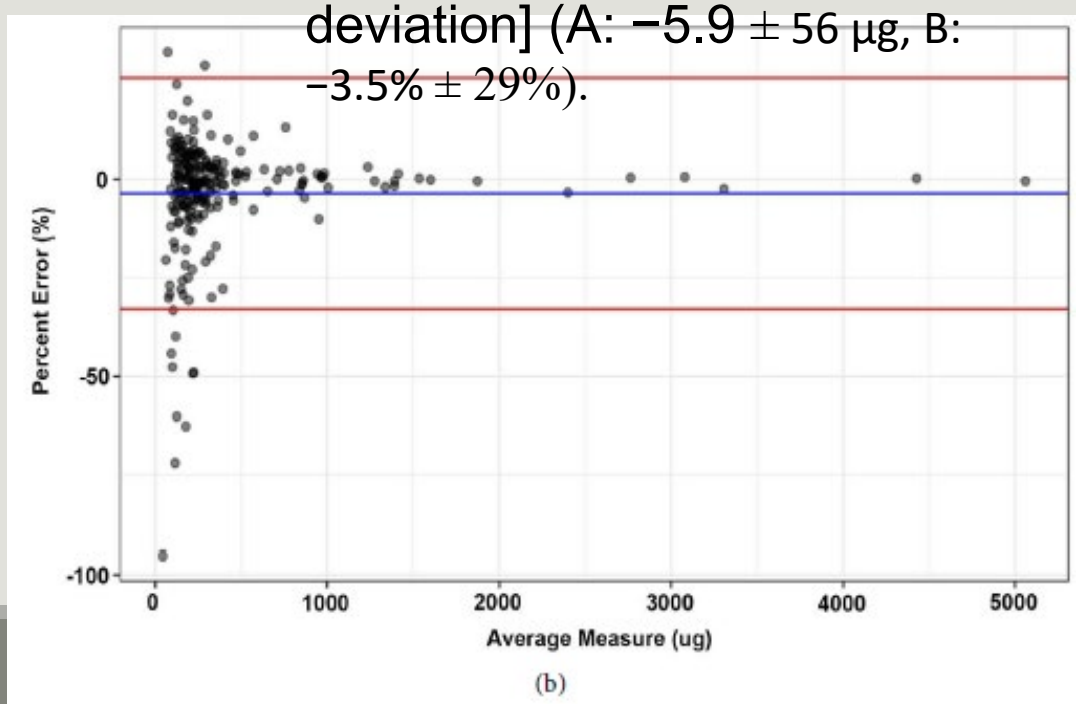


Figure 4. Bland-Altman relationships show the absolute ($M_o - M_{ex}$) (a) and percent (b) error between the two MD determination methods (M_o versus M_{ex}) for all values where $M_{ex} >$ LOD (N = 231). The percent error is calculated using the average of the two measurements. The mean value is defined by the blue line and the 95% limits of agreement are shown in red [mean ± 1.96*standard deviation] (A: −5.9 ± 56 µg, B: −3.5% ± 29%).



(Altman, D.G. and Bland, J.M.,1983)

Concentration Estimate Comparison

- Estimate of PM concentration using a typical gravimetric sample scenario
- Sample duration of 1440 minutes and a flow rate of 1.5 liters per minute was assumed, which is common for exposure and indoor air pollution studies.
- Mean PM MD using standard method was 367 ± 589 µg, and
- Mean PM MD using extraction method was 371 ± 589 µg (diff of 4 µg)
- The standard method yields an appx concentration of 170 ± 272 µg/m^3
- The extraction method yields an appx concentration of 172 ± 273 µg/m^3.

Difference in average approximate concentration is 2 µg/m^3 (1%)

Extraction Method Limitations and Considerations

- Requires field blanks (minimum 10, ideally 10-20%)
- Only tested on PTFE filters with PMP support rings and with wood smoke
- Filters not equilibrated to room temperature prior to extraction or susceptible to tearing, equilibrate to room temperature prior to extraction
- Extracted filters are relatively more electrically charged so anti-static measures should be employed when weighing
- The LoD for the extraction method is 60 ug. This method should not be used during campaigns where filters are thought to be less than 60 ug.
- It is up to the user of this method to decide if the LoA for error (A: −5.9 ± 56 µg, B: −3.5% ± 29%) is acceptable for their application

THANKS!

We wish to thank Kirk Smith and L. Drew Hill from the University of California, Berkeley (UCB) for sharing filters for use in the PM extraction tests and final experiment. We would also like to extend our thanks to Katharine Hammond at UCB for advising on the extraction process and results and Ajay Pillarisetti, Drew Hill, and Madeleine Rossanese for input on the manuscript.

References

This Publication: Garland, C., Delapena, S. and Pennise, D. (2018) An Alternative Technique for Determining Gravimetric Particle Mass Deposition on Filter Substrate: The Particle Extraction Method. Open Journal of Air Pollution, 7, 309-321. https://doi.org/10.4236/ojap.2018.74016

[1] Balakrishnan, K., Sambandam, S., Ghosh, S., Mukhopadhyay, K., Vaswani, M., Arora, N.K., et al. (2015) Household Air Pollution Exposures of Pregnant Women Receiving Advanced Combustion Cookstoves in India: Implications for Intervention. Annals of Global Health, 81, 375-385.

[2] Health and Safety Executive (HSE) (1997) General Methods for Sampling and Gravimetric Analysis of Respirable, Thoracic and Inhalable Aerosols. In: Methods for the Determination of Hazardous Substances (MDHS) Guidance , Health and Safety Executive (HSE).

[3] Rosa, G., Majorin, F., Boisson, S., Barstow, C., Johnson, M., Kirby, M., et al. (2014) Assessing the Impact of Water Filters and Improved Cook Stoves on Drinking Water Quality and Household Air Pollution: A Randomised Controlled Trial in Rwanda. PLoS ONE, 9, e91011. https://doi.org/10.1371/journal.pone.0091011

[4] RTI International. (2008) Standard Operating Procedure for Particulate Matter (PM) Gravimetric Analysis. Environmental and Industrial Sciences Division.

[5] USEPA (2017) Method 5: Determination of Particulate Matter Emissions from Stationary Sources. United States Environmental Protection Agency. https://www.epa.gov/sites/production/files/2017-08/documents/method_5.pdf

[6] Hill, L. D., Pillarisetti, A., Smith, K.R., Delapena, S., Delapena, S., Garland, C., et al. (2015) Air Pollution and Impact Analysis of a Pilot Stove Intervention. Household Energy, Climate, and Health Research Group School of Public Health University of California, Berkeley. http://ehsdiv.sph.berkeley.edu/krsmith/publications/2015/Lao_MoH_Main_report.pdf

[7] Williams, R., Pasley, T., Warren, S., Zweidinger, R., Watts, R., Stead, A. and Claxton, L. (1988) Selection of a Suitable Extraction Method for Mutagenic Activity from Woodsmoke-Impacted Air Particles. International Journal of Environmental Analytical Chemistry , 34, 137-154. https://doi.org/10.1080/03067318808027411

[8] Deming, W.E. (1943) Statistical Adjustment of Data. Wiley, NY (Dover Publications Edition, 1985).

[9] Manuilova, E., Schuetzenmeister A. and Model, F. (2014) Mcr: Method Comparison Regression. R Package Version 1.2.1. https://CRAN.R-project.org/package=mcr

[10] Altman, D.G. and Bland, J.M. (1983) Measurement in Medicine: The Analysis of Method Comparison Studies. Journal of the Royal Statistical Society : Series D (The Statistician), 32, 307-317. https://doi.org/10.2307/2987937